

GA-C22386
(5/97)

HYDROTHERMAL OXIDATION OF NAVY SHIPBOARD EXCESS HAZARDOUS MATERIALS

**ANNUAL STATUS REPORT (CDRL A002)
FOR MAY 1996 THRU APRIL 1997**

**Prepared Under
Contract No. N00014-95-C-0103
for the
OFFICE OF NAVAL RESEARCH**

**Sponsored By
ADVANCED RESEARCH PROJECTS AGENCY**

DISTRIBUTION STATEMENT A
**Approved for public release;
Distribution Unlimited**

DTIC QUALITY INSPECTED 3

19970609 077

 **GENERAL ATOMICS**

GA-C22386
(5/97)

HYDROTHERMAL OXIDATION OF NAVY SHIPBOARD EXCESS HAZARDOUS MATERIALS

**ANNUAL STATUS REPORT (CDRL A002)
FOR MAY 1996 THRU APRIL 1997**

**Prepared Under
Contract No. N00014-95-C-0103
for the
OFFICE OF NAVAL RESEARCH**

**Sponsored By
ADVANCED RESEARCH PROJECTS AGENCY**

This is an informal progress report prepared for the contracting agency. The results and data may be preliminary or tentative and therefore are subject to revision or correction. This report may contain patentable material on which patent applications have not yet been filed and further distribution of this report should not be made without the prior approval of the contracting agency or the contractor.

GA PROJECT 3733



TABLE OF CONTENTS

1. CONTRACT INFORMATION	1
2. DESCRIPTION OF PROGRESS	1
2.1 TASK 100 - PROGRAM DEFINITION	2
2.1.1 Task 110 - Requirements	2
2.1.2 Task 120 – Waste Characterization.....	2
2.2 TASK 200 – SYSTEMS ENGINEERING	2
2.2.1 Task 210 - Trade Studies	2
2.2.2 Task 220 - Safety	2
2.2.3 Task 230 - RAM.....	3
2.2.4 Task 240 - Integrated Process Control	3
2.2.5 Task 250 - Shipboard Integration.....	3
2.2.6 Task 260 - Environmental/Permitting	4
2.3 TASK 300 - DESIGN	4
2.3.1 Task 310 - Conceptual Design.....	4
2.3.2 Task 320 - Preliminary Design.....	4
2.3.3 Task 330 - Final Design.....	6
2.4 TASK 400 - RESEARCH	12
2.4.1 Task 410 - Corrosion	12
2.4.2 Task 420 - Phase Equilibria	15
2.5 TASK 500 - PILOT-SCALE ENGINEERING TESTS	15
2.5.1 Task 510 - EHM Feed/Cold Feed Injection	15
2.5.2 Task 520 - High Pressure Oxidant Supply	16
2.5.3 Task 520 - High Pressure Oxidant Supply	17
2.5.4 Task 540 - Letdown/Solids Separation/Quench	17
2.6 TASK 600 - FABRICATION.....	18
2.6.1 Task 610 - Procurement	18
2.6.2 Task 620 - Equipment Fabrication	19
2.7 TASK 700 - ASSEMBLY & STARTUP	21
2.7.1 Task 710 - Assembly	21
2.7.2 Task 720 - Checkout and Startup Testing.....	21

TABLE OF CONTENTS (CONTINUED)

2.8 TASK 800 - DEMONSTRATION.....	21
2.8.1 Task 810 - GA Tests.....	21
2.8.2 Task 820 - Navy Site Test	22
2.8.3 Task 830 - GA Site Restoration	22
2.9 TASK 900 - PROGRAM MANAGEMENT	22
3. CHANGE IN KEY PERSONNEL.....	22
4. SUMMARY OF SUBSTANTIVE INFO. DERIVED FROM SPECIAL EVENTS	22
5. PROBLEMS ENCOUNTERED AND/OR ANTICIPATED	24
6. ACTION REQUIRED BY THE GOVERNMENT	25
7. FISCAL STATUS	25

1. CONTRACT INFORMATION

ARPA ORDER NO: BAA 94-45 PROGRAM CODE NO: ONR 95431-0162

CONTRACTOR: General Atomics CONTRACT AMOUNT: \$7,715,232.00

CONTRACT NO: N00014-95-C-0103

EFFECTIVE DATE OF CONTRACT: 05/05/95

EXPIRATION DATE OF CONTRACT: 01/31/98

PRINCIPAL INVESTIGATOR: Michael Spritzer

TELEPHONE NO: 619-455-2337

SHORT TITLE OF WORK: Navy HTO or HTO-NSEHM

REPORTING PERIOD: May, 1996 through April, 1997

2. DESCRIPTION OF PROGRESS

This report summarizes progress made during the second year of the DARPA Navy Hydrothermal Oxidation (HTO) program covering the period of May 1996 through April 1997. The report follows the same format as the prior monthly reports and presents a task by task description of activities according to the work breakdown structure (WBS) adopted for the project.

In general, excellent progress has been made on the program during this second year. Activities completed include the remaining HTO systems engineering tasks, supercritical water oxidation corrosion studies at the University of Texas, all pilot-scale HTO engineering tests, and preliminary and final design of the HTO demonstration unit. Procurement and fabrication of equipment for the demonstration unit was started and is nearly complete, and assembly of the demonstration unit was started.

DTIC QUALITY INSPECTED 3

The project schedule has slipped several months due to extended pilot-scale tests and late delivery of procured and fabricated components, but schedule recovery measures have been implemented and we expect to complete the program on schedule. The project budget was actualized at the end of December 1996, and the revised plan is shown in Figure 7-1. Costs continue to track well to the plan and we expect to complete the project within budget. Government planned testing remains to be defined and may require additional funding.

The individual tasks are statused below.

2.1 TASK 100 - PROGRAM DEFINITION

2.1.1 Task 110 - Requirements

- Task complete.

2.1.2 Task 120 - Waste Characterization

- Task complete.

2.2 TASK 200 - SYSTEMS ENGINEERING

2.2.1 Task 210 - Trade Studies

- Task complete

2.2.2 Task 220 - Safety

- The Safety/Hazards Analysis report was completed and issued. The report addresses hazards associated with operation of the DARPA Navy HTO demonstration unit. Mitigating actions to alleviate the frequency and/or severity of occurrence of these hazards are included. The hazards are generally presented separately for each of the HTO unit subsystems, although those hazards common to the entire unit (e.g., ship-wide shock) are grouped under a "General" category. A total of 71 hazards were identified. Following implementation of the specified mitigating factors, all hazards were reduced to

a risk assessment code (RAC) level of either 3 (acceptable with review) or 4 (acceptable without review). This task is now complete.

2.2.3 Task 230 - RAM

- The final Reliability, Availability, and Maintainability (RAM) report was completed and issued. The report contains a detailed list of possible equipment failure modes along with likely causes and consequences of these failures. Mitigating factors to reduce the severity of the failure to an acceptable level are also included. All currently identified failures fall within either Category III (minor injury or minor property damage) or Category IV (no injury or equipment damage but possibility of unscheduled maintenance).

2.2.4 Task 240 - Integrated Process Control

- The Integrated Process Control (IPC) design criteria document was completed and issued. The document defines the DARPA Navy HTO demonstration unit control requirements and hardware. Included are functional requirements for automated process control, data collection and storage, and process alarms, hardware requirements for the push-button control panel, the supervisory computer, the programmable logic controller (PLC), and the system sensors, software requirements for the PLC and supervisory computer software, and interface requirements. This task is now complete.

2.2.5 Task 250 - Shipboard Integration

- Our naval architect subcontractor, M. Rosenblatt & Son (MR&S), continued to provide guidance on issues related to shipboard requirements and demonstration unit/ship interfaces, including information in the following areas: (1) drawings of the proposed layout of the HTO demonstration unit on the hangar deck of a CV or CVN, including routing of piping along the hangar deck, through necessary bulkheads, and along the external ship surface to below water level; (2) details on the electrical distribution system of a CV or CVN along with connection requirements for the demonstration unit; (3) details on attachment of shock mounts to the hangar deck; (4) review of GA's planned pipe routing and supports; (5) details on typical CV and AOE class shipboard

pressure vessels for comparison with potential demonstration unit safety hazards; and (6) details on typical shipboard infrared signature sources for comparison with HTO demonstration unit sources. This task is now complete.

2.2.6 Task 260 - Environmental/Permitting

- The final environmental/permitting report was completed and issued. The report addresses liquid and gaseous effluent emissions requirements for operation of the DARPA Navy HTO demonstration unit during both shoreside testing at GA and during shipboard service. Regulations for discharge into California coastal, Federal, and international waters were considered. For liquid emissions, limits on organic content, pH, inert solids, heavy metals, and temperature are provided. For gaseous emissions, limits on total gaseous emissions are provided. No permits, over and above those already in place for the GA site, are required for shore-based testing, and minimal permitting is expected to be required for shipboard operation. An update of the document is planned for the end of June, 1997, once the Government testing requirements have been defined. If an update is not required, the document will stand as is.

2.3 TASK 300 - DESIGN

2.3.1 Task 310 - Conceptual Design

- **Task complete.**

2.3.2 Task 320 - Preliminary Design

- The preliminary design Technical Data Package (TDP) for the DARPA Navy HTO unit was completed and issued. Documents and drawings comprising the preliminary design TDP are listed in Tables 1 and 2.
- The preliminary design TDP was reviewed by DARPA at a design review meeting held at General Atomics on June 18, 1996.
- Additional information on the progress of the design activity is provided under Task 330, Final Design.

TABLE 1
PRELIMINARY DESIGN TECHNICAL DATA PACKAGE WORD DOCUMENTS

Doc. Number	Title
379001 N/C	Process Description
379003/2	Mass & Energy Balance
379004/A	Oxidant Trade Study
379005 N/C	Reliability, Availability, and Maintainability (RAM) Study
379006/0	Safety/Hazards Analysis Report
379007/0	Alarms and Interlocks List
379008/1	Equipment List
379009 N/C	Air Compressor Trade Study
379010 N/C	Feed/Preheat Trade Study
379011/0	Integrated Process Control Design Criteria
379012 N/C	Reactor Trade Study
379013 N/C	Pressure Letdown Trade Study
379014/A	Equip. Spec. for HP Air Compressor

TABLE 2
PRELIMINARY DESIGN TECHNICAL DATA PACKAGE DRAWINGS

Doc. Number	Title
370001/2	Process Flow Diagram
370002/2	Piping and Instrumentation Diagram
370003/1	General Arrangement
370004/1	Reactor Arrangement
370006/0	Lined Pipe Assemblies
370008/0	Lined Flange Assemblies
370012/1	HX-100 Assembly (Tube-In-Tube)
370020/0	Compact H ₂ O Preheater (H-100)
370022/0	Electrical Enclosure
370025/0	Letdown System
370026/0	Solids Filter w/Screw Auger
370027/0	Quench Chamber
370028/0	Compressor Arrangement Study
370029/0	Ship Location Study
370030/0	Ship Effluent Discharge Routing
370031/0	Ship Sea Water System
370032/0	Shipboard Electrical Interface One-Line Diagram
370033/0	Enclosure

2.3.3 Task 330 - Final Design

Final design of the DARPA Navy HTO unit was completed. The design went through a number of changes as information was gained from pilot testing. A final design review was held on November 8, 1996, although at the time there were a number of major design options that had not been finalized due to a lack of data from ongoing tests. Once pilot plant testing was completed, the final design configuration for the unit was established. The final design includes the following key elements:

- RIX high pressure air compressor.
- "Single-stream" feed system using emulsifier and single high-pressure metering pump for EHM, auxiliary fuel and water.
- Kerosene-fed ignitor to replace electric air preheater for startup.
- Downflow cold feed injection reactor with salt scraper to collect salts at reactor bottom and partial quench to remove metal oxides and prevent reactor plugging.
- High pressure solids filter and lockhopper discharge valve upstream of letdown for on-line heavy metals removal.
- Dual control valves for pressure letdown.
- Seawater quench for final temperature adjustment and pH control.

As the design progressed, the top-level system design documents were periodically updated and reissued. These documents included the process flow diagram (PFD), the piping and instrumentation diagram (P&ID), the electrical-one line diagram, the mass and energy balance (M&EB), the equipment list, and the 3D CAD equipment arrangement model.

The paragraphs below report the progress made in the final design of the various HTO components.

Air Compressor

The equipment specification for the high pressure air compressor was completed and issued. The specification defines the requirements which the compressor must meet, including performance requirements and size constraints. Criteria for performance verification testing were also included. The equipment specification formed the basis for

a request for "best and final" quotations from various compressor vendors. After a thorough review of the offers received, the RIX air compressor was selected for the DARPA Navy HTO unit.

Progress on the compressor design and fabrication was periodically reviewed to assure optimum interface with the HTO design.

EHM Feed

Several alternate configurations for the EHM feed system were investigated in an effort to find a system capable of handling the complex range of Navy EHMs without sorting by Navy personnel. Ultimately, a system whereby the EHM, water, and auxiliary fuel (kerosene) are pumped at low pressure through an emulsifier then to a high pressure pump was selected for the final design of the HTO unit. Specifications for the low pressure pumps, the mixer, and the high pressure pump were developed, and these components were procured. The high pressure pump selected was an Aplex 3-piston pump, with a Clark-Cooper 2-piston pump as a backup. The Clark-Cooper is now planned to be used to supply high pressure quench water.

Because of the difficulties encountered with conventional high pressure pumps during pilot plant testing of paints, several paint pumping alternatives were investigated. Two potentially viable options were identified. The first option uses two syringe pumps connected in parallel to permit continuous operation. A preliminary flow schematic for the dual syringe pump was developed and component sizes were estimated. Layout studies showed that the pump could be accommodated within the existing HTO space envelope. The second option considered was a high pressure slurry pump manufactured by Zimpro. Standard Zimpro pumps have capacities far exceeding HTO requirements, but discussions with Zimpro indicated that Zimpro might be interested in producing a custom design in a smaller size which would fit within the space available in the HTO unit. These options remain as backups, should problems arise with the reference feed system.

Preheater/Ignitor

The preliminary design of the HTO unit included an electric preheater for start up of the SCWO system. Final design of the preheater was completed. However, in an effort to reduce the power consumed by the HTO unit and to conserve space, alternatives to

electric preheat were considered. The most promising concept identified was a direct fired ignitor which would heat high pressure air going to the reactor by burning kerosene.

Design of the fuel-fired ignitor was completed. The ignitor is sized to heat 200 scfm of 4000 psi air from 300°F to 1200°F, using kerosene as the fuel. Calculations were performed to establish required fuel flow rates, line sizes, and component dimensions. Various methods for igniting the fuel-air mixture including glow plugs and spark plugs were considered; a glow plug was selected for the final design. Drawings for the ignitor vessel and components were completed and submitted for fabrication. Specifications for the ignitor pumps, valves, and fittings were developed.

Heat Recovery Heat Exchanger

Design of a tube-in-tube type heat recovery heat exchanger (HRHX) for the demonstration unit was completed, but, to conserve budget, it was decided to defer fabrication of the HRHX. Room has been reserved in the demonstration unit equipment arrangement to allow the HRHX to be added at a later date.

Reactor

Final design of the titanium-lined reactor was completed and reactor fabrication was started. The design includes provisions for internal quench to assist the removal of solids and a mechanical scraper to assist the removal of salt deposits.

The reactor structural material was changed to Inconel 625. Inconel 617 or Haynes 230 would have been preferred because Inconel 625 exhibits a relatively large loss of room temperature ductility with high temperature exposure, but the lead times for Inco 617 and Haynes 230 were excessive. Detailed structural evaluations showed that Inconel 625 was acceptable for the demonstration reactor.

The inside diameter of the reactor tube was increased to 7 inches to provide additional margin in reactor volume while allowing the reactor to be shortened slightly so that it would better fit within the HTO space envelope. As a result of the diameter increase, an ASME code stamp was required for the vessel.

The final reactor design incorporates an O-ring closure for the end flanges. Review of ASME Code requirements confirmed that the reactor flange closure configuration is acceptable under Section VIII. The flange closure construction is similar to that used on pressure vessels commercially manufactured by the High Pressure Equipment Company (HiP) which can be obtained with an ASME Code stamp rated to over 5000 psi.

Thermal/structural analyses using simplified methods were completed to establish the basic sizing of all components. Subsequently, a detailed ANSYS finite element model was developed to enable detailed assessments of temperatures and stresses for various steady state operating conditions. The model showed acceptable operating temperatures in the critical O-ring region as well as acceptable stresses for both the normal operating and hydrotest conditions.

Transient thermal/stress analyses of the reactor for startup and shutdown conditions were performed to confirm that the reactor has adequate margin against fatigue and crack propagation during startup and shutdown. A loss of coolant transient analysis showed that about twenty minutes were available to restart cooling water flow before overheating of the O-ring reactor seals occurred. This time is well within normal restoration time of power and seawater flow aboard ship.

The reactor equipment specification defining fabrication requirements for the reactor was prepared and issued. A bid package consisting of the reactor drawings and the reactor specification was submitted to several potential fabricators (code shops) for quotation. The quotations were received and ERBE engineering was selected to fabricate the vessel, with GA supplying several of the non-code components.

The reactor structural calculations and a preliminary drawing package were submitted to the reactor fabricator, ERBE Engineering, for preliminary review with an ASME code authorized inspector to confirm that all requirements for Code stamping the reactor vessel would be met. No problems were uncovered during this review.

Subsequently, locations for all reactor inlets, outlets, and supports were finalized based on the final location of the reactor within the skid, and the drawing package revised accordingly. The final design drawings and a final calculation package for the reactor were completed and submitted to ERBE for fabrication.

Solids Filter

Several options for solids filtering were explored. Prior to November 8, 1996, the solids filter was located in a hot region downstream of letdown, such that the solids were removed from a hot gas stream at low pressure. Although demonstrated by pilot plant testing, the low pressure hot filter for the HTO unit was large and expensive, and provided no protection of the letdown components from abrasive solids. Options considered included high pressure hot filtering upstream of letdown, and cold filtering following letdown and quench.

Design of the low pressure hot filter continued until November 8, 1996. The design was based on flow through 24 sintered metal filter elements, with pulse blowback. The solids were collected in the bottom of the solids filter vessel, and were removed at the end of a day via an auger arrangement. A specification control drawing and equipment specification for the low pressure solids filter were prepared. Review of corrosion data showed that Inconel 625 was the material of choice for the low pressure solids filter vessel under the design conditions of 300 psi, 1100°F operating temperature, and ten year minimum design life. Other, less costly materials could be used if the design temperature was reduced, or if a shorter design life was acceptable.

Subsequent to November 8, 1996 design of the high pressure hot filter started. The design uses three sintered metal filters housed in a titanium-lined pressure vessel. The filters are cleared by pulse blowback, and the solids are removed from the filter vessel semi-continuously during operation via a lockhopper arrangement.

Because even a small amount of corrosion could plug the pores of the sintered metal filter elements, considerable effort was expended on the element material selection. The GA/UT corrosion tests showed that conventional high-temperature structural materials such as 316SS, C-276, Alloy 625, Alloy 718, and G-30 all exhibit excessive corrosion for at least some feed materials and thus cannot be used for filter elements. The tests showed that several titanium alloys have good corrosion resistance, but many titanium alloys including grades 2 and 7 have relatively low strength at temperatures above about 800°F. The titanium alloys Ti 6242, Ti grade 12, and Ti grade 6 have excellent corrosion resistance combined with good high-temperature strength.

The two largest suppliers of sintered metal filters (Mott and Sintered Metal Krebsorge) were contacted to investigate the feasibility of making the filter elements from any of the high-strength Ti grades. (It was previously known that Ti grade 2 filters were available). It was found that there was little sintering experience with Ti grades other than grade 2, and that powder of the right form and size was generally not available in Ti grades other than grade 2.

A structural analysis of Ti grade 2 filter elements was performed to evaluate possibilities of creep collapse and plastic buckling. Under conservative assumptions, it was estimated that the elements should have a useful life of >1400 hrs, if the operating temperature is limited to less than 800F and the delta-p across them is limited to 50 psi. The elements should not collapse plastically until a delta-p of 385 psi is reached.

Final design of the high-pressure hot solids filter was completed. Calculations were performed to establish the required size and number of filter elements and the size of the filter vessel. The vessel layout was developed to include the effluent in/out passages, cooling channels, and blowback lines. A detailed finite element model of the solids filter was developed for thermal/structural analysis. The analysis confirmed acceptable solids filter stresses and temperatures. Final component drawings were issued for fabrication.

Various options for discharging on-line the solids collected in the solids filter were considered. During pilot-scale testing, the pilot solids filter was fitted with a lockhopper discharge arrangement consisting of two high temperature ball valves in series with a void space in between the two valves to accept a small amount of collected solids. The valves were sequentially cycled to discharge solids as they were collected. This arrangement worked adequately for proof of principle testing, but over time the ball valves began to leak and jam. After extensive searches for alternatives, a "rotating disc valve" manufactured by the Everlasting Valve Company was identified as a potentially longer-lasting valve for a lockhopper design.

The basic Everlasting Valve is relatively large however, so a considerable effort was spent with the manufacturer to identify a compact configuration which would be acceptable for the DARPA Navy HTO unit. Ultimately, a relatively compact "unitandem" configuration was developed which meets the space constraints.

Liquid phase solids filters for cold letdown were investigated. New Logic International, manufacturer of the VSEP vibrating membrane filter, was visited to discuss details of the VSEP filter design and its operating parameters. Another candidate filter, a centrifugal membrane filter manufactured by SpinTek, was also investigated. Both of these filters appear to be viable options for the cold letdown solids filter.

Information on an advanced ion exchange resin for heavy metals capture from seawater was obtained. The resin was developed for use on Trident submarines. It was tested recently on a submarine and shown to effectively extract heavy metals from seawater.

Layout studies were performed to determine whether cold letdown could be accommodated within the DARPA-mandated 8' x 9' x 10' HTO space envelope. Sizes for the major cold letdown components (quench pump, liquid phase solids filter [assumed to be VSEP], air-liquid separator, and ion exchange bed) were estimated, and models of the components were incorporated into the 3D HTO CAD model. The studies show that the cold letdown components could fit in the available space.

Quench

Final design of the quench vessel was completed, and the unit submitted to Koch for fabrication. The quench vessel consists of a 6" sch 40 pipe housing a spray nozzle at one end (for cocurrent seawater injection into the gas stream) and five downstream static mixers. The material of construction is 316L.

2.4 TASK 400 - RESEARCH

2.4.1 Task 410 - Corrosion

- Corrosion testing of a variety of metals under Navy HTO conditions was performed at the University of Texas (UT), and at Los Alamos National Laboratory (LANL).

UT Corrosion Testing

The UT corrosion tests investigated the corrosion resistance of a variety of metals, including nickel alloys, titanium alloys, and noble metals. Four different acid media were tested, each simulating the worst-case effluent products resulting from destruction of

specific Navy EHMs. These media were sulfuric acid/water/oxygen ($H_2SO_4/H_2O/O_2$), phosphoric acid/water/oxygen ($H_3PO_4/H_2O/O_2$), hydrochloric acid/water/oxygen ($HCl/H_2O/O_2$), and hydrofluoric acid/water/oxygen ($HF/H_2O/O_2$). Test were conducted at discrete temperatures of 350°C, 450°C, and 625°C, for durations of 5, 20, and 100 hrs.

- The 36 planned corrosion tests were completed. Corrosion rates were determined for all materials tested based on precision weight loss measurements. Selected coupons, primarily titanium alloys, were then subjected to further investigation by scanning electron microscopy (SEM), energy dispersive X-ray (EDX), and microhardness (MHD) analyses.
- GA engineering staff visited UT to review the corrosion data and to provide guidance on data interpretation. In general, the high nickel alloys such as Hastelloy C276 showed varying degrees of relatively poor performance (as exhibited by large metal loss and/or pitting) for most media/temperature combinations. Niobium and silver were almost completely destroyed by one or more media/temperature condition. Platinum and gold fared somewhat better for most conditions, but were severely attacked by 350°C chloride. Titanium grades 2 and 7 performed very well, with generally low corrosion rates (<10 mills per year) for most media/temperature combinations except 450°C phosphate. The high strength Ti grade 12 and 6242 alloys also performed very well, although these materials were added later in the program and thus were not tested for 350°C phosphate, or for sulfate. No embrittlement of titanium was observed.
- The thesis of the principal investigator, Andrew Robertson, was completed and submitted for approval. The thesis contains the corrosion test description and data and will be included as an attachment to the final corrosion test report, which is in progress.

LANL Corrosion Tests

While the UT corrosion tests were performed at three discrete temperatures with a large variety of materials, the LANL corrosion tests were directed toward deriving corrosion rate data for titanium over a range of temperatures from 20 to 600°C.

- Originally, it was planned to perform the LANL corrosion testing using test media compositions essentially identical to those anticipated for worst-case EHM processing in the demonstration unit. For H_2SO_4 testing for example, the planned media composition was 50% N_2 , 37% H_2O , 3.0% O_2 , 9.7% CO_2 , and 0.3% H_2SO_4 . During the test set up phase, LANL encountered difficulties delivering the required percentage of nitrogen. Therefore, CO_2 was substituted for nitrogen since it could be pumped relatively easily in liquid form.
- High corrosion of Grade 2 and Grade 7 titanium was observed during the initial H_2SO_4 corrosion tests. This was believed to be related to the excess CO_2 used in the test. Therefore, a series of tests was performed to shed further light on the effect of the excess CO_2 . These tests included the following: (1) a second high CO_2 test to confirm the earlier results, (2) a test without CO_2 to investigate corrosion in the absence of CO_2 , and (3) a test with a CO_2 concentration more typical of HTO conditions. The early results for the tests showed excessive corrosion (in the inches/yr range) for essentially all of the tests. Upon further discussion with LANL, an error was found in the corrosion rate calculations. The revised corrosion rates for the typical and no CO_2 cases now show corrosion rates of 150-300 mil/yr, within acceptable levels especially given the relatively small percentage of feeds containing sulfur or other highly corrosive feeds. The high CO_2 test results still show an elevated corrosion rate, but the high CO_2 levels are not representative of HTO operation.
- Four of the eight planned tests were completed. Progress on the other tests was delayed due: 1) to a delay in receipt of further DARPA funding, 2) the move of the LANL HTO laboratory to an adjacent facility, and 3) the replacement of the piston-type waste feed pump with a syringe pump. These issues were resolved and testing was resumed with initial efforts directed toward generation of reactor axial temperature profiles. This testing effort is now scheduled to be completed by the end of July, 1997. While all material selections have already been made, this corrosion data will provide additional information about potential failures and material upgrades required to overcome corrosion from actual EHMs.

2.4.2 Task 420 - Phase Equilibria

Phase equilibria testing was performed at LANL in two major areas. The Group I experiments investigated the phase behavior of water/gas/acid and water/gas/salt mixtures at near critical conditions. The Group II experiments investigated the phase behavior of water/gas/acid mixtures at nominal operating conditions of 625°C and 4000 psig.

- The final Phase Equilibria test report was prepared by LANL, and the report was issued. In general, the conclusions from this work are that no unusual or unexpected salts precipitation or phase separation is likely to occur for the range of conditions expected with Navy EHMs. The results also provided insight about the upward shift in critical point temperature (and the corresponding effects on corrosion) when acids and diluent gases are added to water.
- The Group I acidic systems studied were: (1) 0.3% H₂SO₄/balance H₂O, (2) 0.5% HCl/balance H₂O, and (3) 0.5% HCl/10% O₂/balance H₂O. The measured critical points for these mixtures were 406, 395, and 410°C, respectively. The Group I salt systems studied were: (1) 1% Na₂SO₄, (2) 0.1% Na₂SO₄, (3) 0.01% Na₂SO₄, and (4) 0.1% NaHSO₄. For all of these mixtures, salt precipitation began to occur over a very narrow temperature range of 390 to 394°C.
- The Group II systems studied were: (1) 0.5% HCl/37% H₂O/53% N₂/9.5% CO₂, (2) 0.3% H₂SO₄/37% H₂O/53% N₂/9.7% CO₂, and (3) 2.0% H₃PO₄/37% H₂O/53% N₂/8.0% CO₂. Phase behavior plots for each of these systems are presented in the test report.

2.5 TASK 500 - PILOT-SCALE ENGINEERING TESTS

2.5.1 Task 510 - EHM Feed/Cold Feed Injection

Cold feed injection (CFI) testing of the baseline EHM feeds was completed. Paint, photographic fluid simulant, and molybdenum disulfide (MoS₂) lubrication oil were

successfully processed by CFI during the period. Previous tests had demonstrated CFI processing of all other Navy EHMs. Some highlights of the testing are discussed below.

- Initial paint testing required use of a syringe pump because the high viscosity of the paint precluded use of the standard DARPA SCWO pilot plant feed system. Information gained from pilot plant paint processing was transferred to the design of the demonstration unit.
- Internal quench was used as the salts handling method for processing photographic fluid simulant containing high loadings of Na_2SO_3 , K_2SO_3 , and $(\text{NH}_4)_2\text{SO}_3$. The effluent from photographic fluid simulant processing was very slightly yellowish-green. The color was attributed to corrosion of the Hastelloy C276 piping connecting the reactor outlet to the cooldown heat exchanger by sulfuric acid in the effluent stream.
- MoS_2 lubrication oil was successfully processed at up to 77 weight per cent of oil in kerosene, significantly greater than the 10% level stipulated by the baseline feed table. TOC levels were typically near zero.
- During one MoS_2 lubrication oil test the oil in kerosene ratio was raised to 85 wt-% after several hours of continuous run time at 77 wt-%. At that time, ignition was lost in the upper reactor zone, apparently due to insufficient breakup of the oil caused by the high oil viscosity. When ignition was lost, water flow was reduced; when ignition was regained, an over-temperature occurred which resulted in failure of the reactor outlet piping. No personnel were injured as a result of the event, and no damage to the reactor itself occurred. Investigation determined that the failure was due the formation of a low-melting eutectic which resulted in overpressurization of a Hastelloy C-276 fitting. Procedures were implemented to prevent recurrence of the problem. The reactor exit piping was repaired, the reactor was reinstalled, and further testing was completed without incident.

2.5.2 Task 520 - High Pressure Oxidant Supply

Pilot plant testing was completed using a leased air compressor for the high pressure oxidant supply. Task activities with this compressor are complete. Activities associated

with the demonstration unit high pressure compressor package are discussed in Sections 2.3.2 and 2.6.1.

2.5.3 Task 520 - High Pressure Oxidant Supply

Testing associated with mechanical salts removal (Task 530) and letdown/solids separation/quench (Task 540) were combined into an integrated test program. The results are discussed together in the following section.

2.5.4 Task 540 - Letdown/Solids Separation/Quench

Installation and shakedown of all pressure letdown, solids separation, and quench test equipment in the DARPA SCWO pilot plant was completed and integrated testing was performed. Approximately 27 test runs were completed (described in detail in prior monthly reports) which verified the demonstration unit design approach for salts (collection in the reactor with subsequent flushing), as well as the approach for solids (removal in a dry solids separator). Hot pressure letdown and quench concepts were also verified. Major components installed and tested include: (1) the test reactor, equipped with an internal salts scraper; (2) a hot pressure letdown system consisting of three parallel capillaries for partial pressure letdown and a Kammer control valve for final pressure letdown and flow control, (3) a solids separator incorporating three sintered metal filter elements and a blowback system for solids removal, and (4) an internal quench system for temperature control of the process effluent prior to release. Additionally, a quench water recirculation system was installed to simulate seawater quench while avoiding the need to collect and discharge large quantities of process water.

On the basis of the pilot-scale engineering tests, the design basis for the demonstration unit was confirmed as follows:

- cold feed injection of all EHM feeds
- single feed system with emulsifier
- salts scraper to transport salt to bottom of reactor
- partial quench to transport paint pigments and other metal oxides from reactor and to preclude plugging of the reactor bottom for salts or solids

- high pressure hot filtration of paint pigments and metal oxides
- high temperature pressure letdown
- seawater quench to reduce effluent temperatures and to adjust effluent pH

2.6 TASK 600 - FABRICATION

2.6.1 Task 610 - Procurement

Procurement of components for the HTO demonstration unit was started and nearly completed through the period at GA and EWT. Orders were placed for all major components, including the supervisory computer, the PLC components, the high pressure air compressor, various high and low pressure pumps, the in-line feed mixer, the titanium sintered metal filter elements, the solids filter discharge valve, the quench unit, and the HTO enclosure. A cooling tower, required for shore-based testing at GA, was also ordered, as were various other miscellaneous valves, fittings, piping, tubing, and instruments. Many of the ordered items have been received at GA. Highlights of the procurement activity are as follows:

- Following extensive discussions with several high pressure air compressor vendors conducted last year to optimize their compressor packages for the DARPA Navy HTO application, "Best and Final" offers for the HTO demonstration unit high pressure air compressor package were received from six vendors. The most favorable proposals were received from RIX Industries, Henderson International, and Dresser-Rand. The Dresser-Rand compressor was the most compact unit, but was high in cost. The Henderson was lowest in cost, but was larger than either the RIX or the Dresser-Rand. Studies of alternate HTO equipment arrangements utilizing the Henderson compressor were completed and showed that there was a high risk of exceeding DARPA's specified HTO demonstration unit space envelope if the Henderson compressor were used. DARPA was requested to allow an increase in the size of the HTO unit to accommodate the less expensive but larger Henderson compressor, but this request was denied. Therefore, it was decided to proceed with purchase of the RIX compressor.

- Following the decision to proceed with purchase of the RIX compressor, negotiations between RIX and GA on the compressor specifications and cost were completed. Subsequently, the purchase order for the compressor was placed.
- An extensive survey of pump manufacturers was conducted which identified several alternate vendors capable of supplying pumps suitable for the HTO demonstration unit. Data on performance, size, cost, and delivery time for several candidate pumps were collected and evaluated. Detailed specifications for the two high pressure (4000 psi) pumps were prepared, and orders were placed for these pumps with APLEX Industries and Clark-Cooper. A drum pump manufactured by IRIS industries was selected and ordered for the low pressure auxiliary fuel pump, and a progressive cavity pump manufactured by SEEPTEX was selected and ordered for the low pressure EHM pump.
- A survey of mixers, maceraters, and emulsifiers was conducted to identify alternatives for the mixer for the single stream feed option. A two-stage Silverson mixer/emulsifier was determined to offer the best combination of performance, size, cost, and delivery for the DARPA Navy HTO application. A single-stage rental unit was procured for pilot plant testing.

2.6.2 Task 620 - Equipment Fabrication

Fabrication of all major components for the DARPA Navy HTO demonstration unit was started including the high pressure air compressor, the high pressure feed pumps, the ignitor, the reactor, the solids filter, the quench unit, the reactor skid, and the shoreside testing cooling tower. Several of these items have been completed, but several are late. Concerns persist over the quality of work being provided by a number of vendors. The status of key components is as follows:

- Fabrication of the HP air compressor was completed at RIX and shakedown testing was started. GA personnel visited RIX on several occasions to monitor the progress of the design and to review interfaces between the compressor and the HTO unit to assure that all requirements were addressed. An

acceptance test plan for the compressor was prepared by RIX, reviewed by GA, and approved. During the initial shakedown testing of the compressor several startup problems occurred. These problems included overheating of a crankcase bearing, failure of an oil lubricator O-ring, leakage of a cylinder liner closure plug, and failure of the last stage wrist pin bearing. These problems are in the process of being resolved. It is expected that the acceptance test will be completed in early June and the compressor delivered to GA shortly afterwards.

- Fabrication of the Aplex high pressure pump was completed and the pump delivered to GA. However, upon initial testing the pump was found not to meet flow performance requirements. Efforts were initiated with representatives from Aplex to improve the efficiency of the pump. Aplex installed new valves in the pump, but these did not significantly improve performance. Aplex then fabricated new, larger pistons for the pump. After these pistons were installed and the drive gear ratio adjusted, the required pump flow was achieved. However, a new problem has emerged with noise under load. Aplex has been contacted to resolve the problem.
- Fabrication of the Clark-Cooper pump was started, but various problems, including a move of the Clark-Cooper facility and machining difficulties delayed completion. GA increased its efforts to expedite this item. The pump will be tested at the factory before it is shipped to GA. This test is expected to occur in early June.
- Fabrication of the ignitor vessel and internal components was started and is nearly complete.
- Fabrication of the reactor was started and is well underway. The reactor vessel, tube nut, and flange materials were received and ultrasonically inspected. Machining of these components is in progress. The reactor vessel was trepanned. The reactor titanium liner and the salt scraper have been rolled and welded. Material for the reactor titanium inserts was received and ultrasonically inspected. Machining is well underway.

- Fabrication of the solids filter was started and is well underway. The solids filter vessel, tube nut, and flange materials were received and ultrasonically inspected. Machining of these components is in progress. The solids filter vessel was trepanned. The solids filter titanium liner was rolled and welded. Material for the solids filter titanium inserts was received and ultrasonically inspected. Machining is well underway. Fabrication of the titanium filter elements is in progress, as is fabrication of the Everlasting solids filter discharge valve.
- Fabrication of the titanium-lined tubing, flanges, and cooled seal rings was started and is well underway.
- Fabrication of the HTO enclosure skid was completed and the skid delivered to GA.

2.7 TASK 700 - ASSEMBLY & STARTUP

2.7.1 Task 710 - Assembly

- Facility modifications to support operation of the HTO unit were started and nearly completed. A new concrete pad for the HTO cooling tower was poured and the cooling tower installed on the pad. Plumbing and electrical hookup are in progress. A new 800 amp 480 volt electrical circuit to supply power to the HTO unit was installed.
- Assembly of the HTO unit was started.
- Programming of the PLC and supervisory computer was started.

2.7.2 Task 720 - Checkout and Startup Testing

- No activity

2.8 TASK 800 - DEMONSTRATION

2.8.1 Task 810 - GA Tests

- No activity.

2.8.2 Task 820 - Navy Site Test

2.8.3 Task 830 - GA Site Restoration

- No activity

2.9 TASK 900 - PROGRAM MANAGEMENT

- Budget and cost control activities continued throughout the reporting period.
- Technical exchange between the GA Navy HTO project team and the Stone & Webster (SWEC)/GA HTO project team continued on a broad range of topics. In collaboration with the GA Navy HTO project team, the SWEC/GA HTO project team developed an alternate heavy metals removal concept for the SWEC/GA design. The concept utilizes advanced membrane filtration followed by selective ion exchange.
- Incremental funding was received from ONR, bringing the contract to full value. At year end, following completion of the pilot-scale engineering tests and verification of the final design configuration, the budget was actualized through December, 1996 and the planned spending profiles were revised for each task for the balance of the contract. As part of this rebudgeting effort, cost-reduction measures were incorporated to better match the remaining work and to add assurance of being able to complete the project on schedule and within budget.
- On April 7, 1997, notification was sent to the Governments' contracting officer that spending had reached 75% of the obligated funding limit.

3. CHANGE IN KEY PERSONNEL

- No significant staffing problems or changes are anticipated.

4. SUMMARY OF SUBSTANTIVE INFORMATION DERIVED FROM SPECIAL EVENTS

- On June 11, 1996, GA participated in the second DARPA Navy HTO joint contractor's briefing held at the offices of System Planning Corporation in

Arlington, Virginia. One of the major objectives of the meeting was to brief representatives of the Navy responsible for transferring HTO technology to shipboard use following demonstration of the technology under the DARPA Navy HTO program. DARPA presented an overview of the HTO program including preliminary plans for shoreside demonstration testing. GA's presentation included discussions of the status of the GA HTO demonstration unit design, results of recent pilot-scale experiments, general HTO technology issues, ship interface issues, and operation and maintenance issues. Potential extension of the HTO technology to Navy wastes other than liquid EHMs was also briefly discussed.

- On June 18, 1996, a review of GA's preliminary design of the DARPA Navy HTO demonstration unit was held at GA's facilities in San Diego. Those in attendance included representatives of DARPA, ONR, the Navy, and GA. Details on the completed preliminary design were presented, and a presentation handout was provided to all participants. Excellent input was received from all attendees.
- Subhas Sikdar, DARPA Navy HTO Project Advisory Board Member, visited GA and was briefed on the program status. At the suggestion of DARPA, Mike Rigden of the Institute for Defense Analysis visited GA and was briefed on the program status and given a tour of the DARPA/SCWO Pilot Plant. Both Dr. Sikdar and Dr. Rigden were favorably impressed with the program status and accomplishments.
- A project review meeting was held on November 8, 1996. While the meeting served as a final design review, there were a number of major design options that had not been finalized due to a lack of data from ongoing tests. These options were discussed at length with respect to technical and programmatic risks for near-term decisions (with or without additional data). Following a thorough review of the issues, it was concluded that GA should: 1) continue testing for several weeks to help verify that hot-letdown and dry solids-separation can be made to work; 2) finish the design and procure equipment based on hot-letdown and dry solids-separation using data available in several weeks; 3) test hot-letdown and dry solids-separation during GA demonstration

testing; and 4) if hot-letdown and dry solids-separation cannot be made to work, adopt cold-letdown and wet solids-separation and test with the GA prototype unit during Government demonstration testing. Subsequently, pilot - scale tests of hot letdown and dry solids collection continued through December during which the reference technical approach was verified as sound.

- Plans were made to support the third DARPA Navy HTO joint contractor's briefing scheduled for May 22, 1997. The meeting will focus on the Government's test program and initial discussions with the test contractor A.D. Little.

5. PROBLEMS ENCOUNTERED AND/OR ANTICIPATED

The initial testing of the high-temperature letdown and solids separation concept did not produce acceptable results. As a result, additional effort was required. This issue was reviewed with Government technical personnel during the Project Review meeting on November 8, 1996. It was concluded that we should continue to pursue hot letdown and dry solids collection within the current budget and schedule. Pilot -scale tests of hot letdown and dry solids collection continued through December during which the technical approach was verified as sound. The design was subsequently frozen, and cost-reduction measures implemented and the program rebudgeted to better match the remaining work and to add assurance of being able to complete the project on schedule and within budget.

6. ACTION REQUIRED BY THE GOVERNMENT

- Incremental funding was received from ONR bringing the contract to full value.
- Action is required to define the Governments' test program and to identify potential cost and schedule impacts, if any.

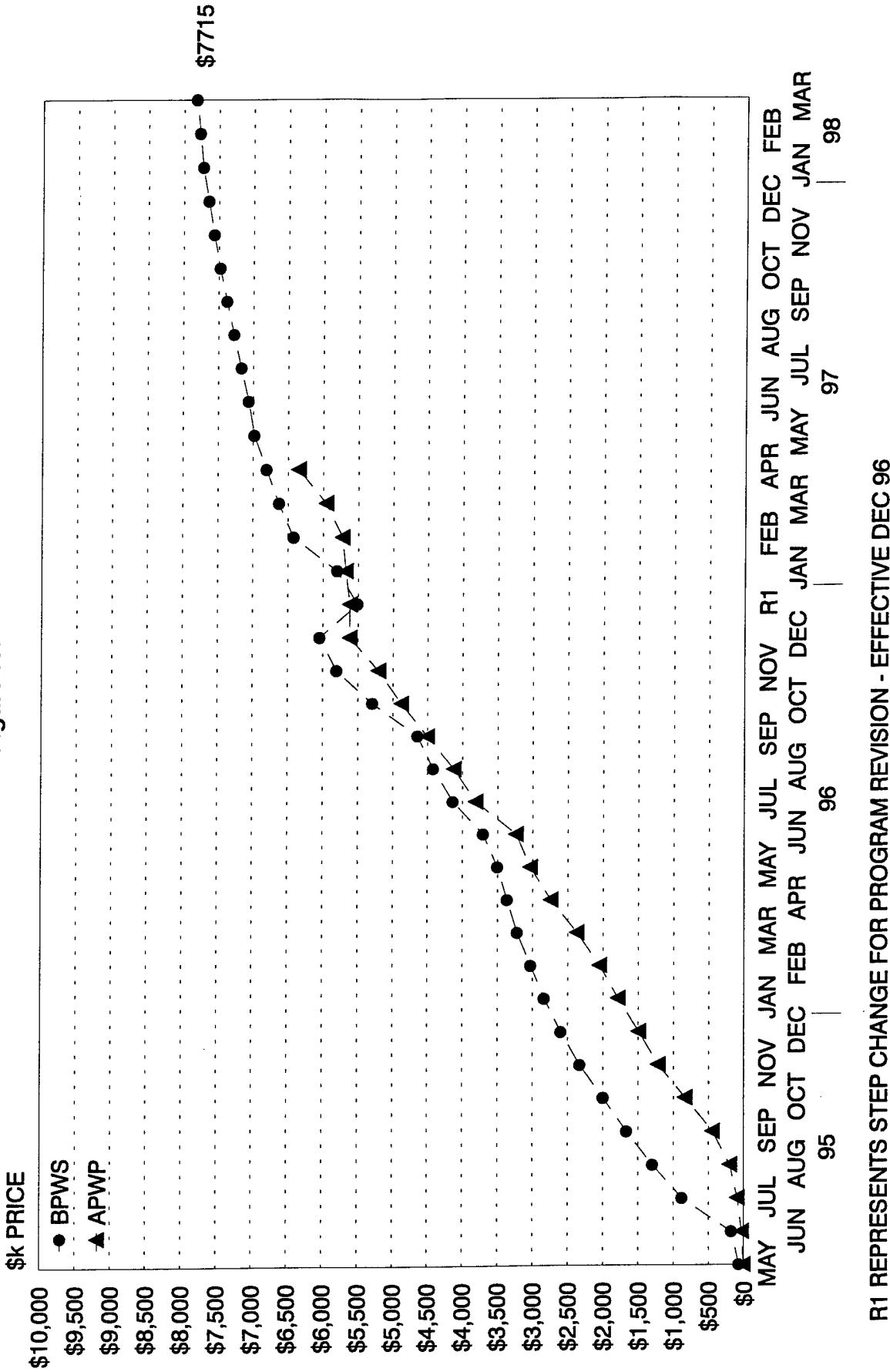
7. FISCAL STATUS

- The amount currently provided on the contract is \$7,715,232. Expenditures and commitments through the end of April 1997 are \$6,348,925. The funds required to complete the work are \$1,366,307. All figures include fee. Figure 7.1 shows the overall program cost status, including cost actualization through December 1996. Spending and schedule variances persist due to procurement delays for major hardware components. Figure 7.2 shows the project schedule and the status of project milestones. Spending is expected to catch up with the plan in one or two months. Schedule recovery actions are being implemented. We expect to complete the contract within budget.

DARPA NAVY HTO - PROJECT SUMMARY

CUMMULATIVE COST WITH FEE THRU 4/25/97

Figure 7.1



DARPA NAVY HTO WBS, SCHEDULE, AND MILESTONES

Figure 7.2

